

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification<sup>5</sup> :</b> <b>C07C 43/11, C11D 1/72</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 94/11330</b> <b>(43) International Publication Date:</b> 26 May 1994 (26.05.94)
<b>(21) International Application Number:</b> PCT/SE93/00966 <b>(22) International Filing Date:</b> 12 November 1993 (12.11.93)  <b>(30) Priority data:</b> 9203478-4 19 November 1992 (19.11.92) SE  <b>(71) Applicant (for all designated States except US):</b> BEROL NOBEL AB [SE/SE]; S-444 85 Stenungsund (SE).  <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only) :</b> DAHLGREN, Lennart [SE/SE]; Mjölmarvägen 4, S-444 95 Ödsmål (SE). BERGSTRÖM, Karin [SE/SE]; PL 3842, S-442 95 Kungälv (SE).		<b>(74) Agent:</b> ANDERSSON, Rolf; Berol Nobel AB, S-444 85 Stenungsund (SE).  <b>(81) Designated States:</b> CA, FI, JP, NO, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ALKOXYLATE OF 2-PROPYL HEPTANOL AND USE THEREOF  <b>(57) Abstract</b>  The invention relates to an alkoxylate of the general formula (I): $C_5H_{11}CH(C_3H_7)CH_2O(B)_r(C_2H_4O)_pH$ , wherein B is an alkyleneoxy group having 3-4 carbon atoms, p is 1-10 and r is 1-6. The alkoxylate may be included as a surface-active component in compositions for cleaning textile materials.		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LJ	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

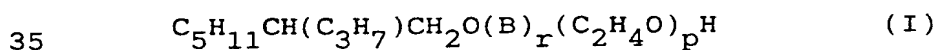
## ALKOXYLATE OF 2-PROPYL HEPTANOL AND USE THEREOF

The present invention relates to an alkoxylate of 2-propyl heptanol. The alkoxylate exhibits high detergent power on textile materials and low foaming compared with similar compounds having a hydrophobic group of approximately the same size and approximately the same HLB-value. The alkoxylate may advantageously be used as a surface-active component in detergent compositions for textile materials.

It has long been known to alkoxylate alcohols for obtaining non-ionic surface-active compounds. These compounds have been used in detergent compositions because of their wetting and dispersing properties. In a number of applications, alkoxylates of  $C_{8-11}$  alcohols have however been found to be too high-foaming and/or not to have the desired detergent power. For example, ethoxylates based on branched  $C_8$  alcohols often exhibit acceptable foaming but too low a detergent power, whereas ethoxylates based on straight or branched alcohols having a larger hydrocarbon chain often show an acceptable surface activity but too high foaming. Thus, there is a need for new alkylene oxide adducts with an improved ratio of foaming to detergent power.

It has now been found that an alkoxylate based on 2-propyl heptanol has good detergent and wetting properties as well as low foaming as compared with other alcohols having substantially the same chain length. In addition, it has been found that the alkoxylate is easily degradable and has a surprisingly low biotoxicity. In tests, no skin-irritant effect has been noted.

The alkoxylate according to the invention can be illustrated by the formula



wherein B is an alkyleneoxy group having 3-4 carbon atoms, p is 1-10 and r is 1-6. Preferably, p is 2-8 and r is 1-4. In these compounds, the hydrophobic properties of the hydrocarbon chain have been enhanced by adding hydrophobic alkyleneoxy groups closest to the alcohol. The compounds have a good detergent power on textile materials while at the same time showing slightly lower foaming in relation to compounds having a hydrophobic group of approximately the same hydrophobicity and approximately the same HLB-value.

The alkoxyates according to the invention described above can be prepared by adding in a conventional manner in the presence of a conventional alkali catalyst, such as potassium hydroxide or sodium hydroxide, the above-mentioned amounts of alkylene oxide to 2-propyl heptanol, which is a so-called Guebert alcohol. According to a preferred mode of execution, the addition of ethylene oxide is performed using a conventional catalyst which gives a narrower distribution of added ethylene oxide than any alkali catalyst, such as NaOH or KOH. Thus prepared alkoxyates according to the invention have very low foaming. Examples of conventional catalysts giving a narrow distribution of added alkylene oxide are  $\text{Ca(OH)}_2$ ,  $\text{Ba(OH)}_2$ ,  $\text{Sr(OH)}_2$  and hydrotalcite. The reaction is preferably conducted in the absence of free water to reduce the amount of by-products and usually at a temperature of 70-180°C.

Textile-cleaning compositions including the alkoxyate according to the invention may also contain other surface-active compounds, such as anionic ones. Examples hereof are alkyl sulphate, alkyl ether sulphate, alkyl benzene sulphonate,  $\alpha$ -olefin sulphonate and alkyl glyceryl sulphonate. Other commonly occurring components are solutising additives, complexing agents and/or pH-adjusting agents, enzymes, bactericides and perfumes. The compositions are usually aqueous and in the form of emulsions, microemulsions or solutions.

The invention will be further illustrated by the following Examples.

#### Example 1

An alkoxyate according to the invention are prepared by alkoxyating 2-propyl ethanol with the amounts of alkylene oxide appearing from the Table below in the presence of potassium hydroxide as catalyst. For reference purposes, two alkoxyates were prepared using a C<sub>9-11</sub> alcohol (Dobanol 91 Shell) as hydrophobic ingredient. The resulting products were analysed and structurally determined by gas chromatography and mass spectrometry. The turbidity points were measured in water or monobutylether diethylene glycol. The following results were obtained.

Table 1

Compound	Alcohol	Mole of alkylene oxide/mole of alcohol	Catalyst	Turbidity point	
				Water	BDG
1	2-propyl heptanol	4 PO+6 EO <sup>1)</sup>	KOH	25	-
A	C <sub>9-11</sub> alcohol	4 EO	KOH	-	62
B	C <sub>9-11</sub> alcohol	6 EO	KOH	56	-

EO = ethylene oxide; PO = propylene oxide,

BDG = monobutylether diethylene glycol

<sup>1)</sup> PO added first

#### Example 2

The foaming properties of the alkoxyates reported in the following Table were measured according to Ross-Miles ASTM D 1173-53. The following results were obtained.

Table 2

Compound	Foam height, cm	
	0 min	5 min
1	83	12
A	80	20
B	95	30

The compound according to the invention has equivalent or slightly lower foaming as compared with compounds A and B.

**Example 3**

Washing tests were carried out in a Terg-O-Tometer on pigment-soiled cotton and cotton/polyester. Washed-away soil was thereafter determined by conventional reflectance measurement. The following results were obtained.

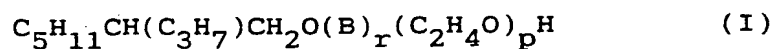
Table 3

Compound	Washed-away pigment soil, %		
	Cotton	Cotton/polyester	
	40°C	40°C	60°C
1	78	73	66
A	78	65	52

From these results it appears that the compound according to the invention all in all has higher detergent power than the reference compound. From Example 2 also appears that the compound according to the invention has slightly lower foaming than the reference compound.

## C L A I M S

1. An alkoxylate as claimed in claim 1 or 2, c h a r -  
5 a c t e r i s e d by having the general formula



wherein B is an alkyleneoxy group having 3-4 carbon atoms,  
10 p is 1-10 and r is 1-6.

2. An alkoxylate as claimed in claim 1, c h a r a c -  
t e r i s e d in that p is 2-8 and r is 1-4.

- 15 3. The use of a compound as claimed in claim 1 or 2 in  
a detergent composition for textile materials.

20

25

30

35

1  
INTERNATIONAL SEARCH REPORT

International application No.  
PCT/SE 93/00966

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C07C 43/11, C11D 1/72

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C07C, C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

CA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB, A, 2145726 (DIVERSEY CORPORATION), 3 April 1985 (03.04.85) --	1-3
A	CH, A5, 674358 (OUHADI TRAZOLLAH ET AL.), 31 May 1990 (31.05.90) --	1-3
A	EP, A1, 0046582 (CONOCO INC.), 3 March 1982 (03.03.82) --	1-3
A	US, A, 3567784 (WILLIAM T. TSATSOS ET AL.), 2 March 1971 (02.03.71) -----	1-3

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	<p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&amp;" document member of the same patent family</p>
--	---

Date of the actual completion of the international search

22 February 1994

Date of mailing of the international search report

25 -02- 1994

Name and mailing address of the ISA/  
Swedish Patent Office  
Box 5055, S-102 42 STOCKHOLM  
Facsimile No. +46 8 666 02 86

Authorized officer

Eva Johansson  
Telephone No. +46 8 782 25 00



**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

28/01/94

International application No.  
PCT/SE 93/00966

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
GB-A-	2145726	03/04/85	DE-A-	3431156	02/05/85
			FR-A-	2550959	01/03/85
CH-A5-	674358	31/05/90	AU-B-	595060	22/03/90
			AU-A-	7735987	03/03/88
			DE-A-	3727793	10/03/88
			FR-A-	2603277	04/03/88
			GB-A,B-	2194536	09/03/88
			JP-A-	63063630	22/03/88
			LU-A-	86979	02/03/88
			NL-A-	8702024	16/03/88
			SE-A-	8703301	01/03/88
EP-A1-	0046582	03/03/82	SE-T3-	0046582	
			CA-A-	1157052	15/11/83
			JP-C-	1555089	23/04/90
			JP-A-	57042646	10/03/82
			US-A-	4302613	24/11/81
US-A-	3567784	02/03/71	NONE		

2003-903407/82	A25 B07 C07 D25 E17 F06 G02 (A97 D18 D21 F09 G03)	BADI 2002.04.26	A(10-E8A, 12-W12C) B(4-C3C, 10-E4C, 12-M9) C(4-C3C, 10-E4C, 12-M9) D(7-B, 8-B13, 11-A3A) E(10-HID, 11-D, 11-F) F(3-C5, 5-A6D) G(2-A3) N(4-A, 7-B, 7-D) .3
BASF AG 2002.09.18 2002-1043360(+2002DE-1018754) (2003.11.06) C07C 43/11, 41/03, C11D 1/72	*WO 2003091191-A1 New and known 2-propyl-heptan-1-ol alkoxylates, used e.g. in cleaning, coating, adhesive, textile treatment, cosmetic, pharmaceutical or plant protection formulations, contain ethylene oxide and other alkylene oxide groups (Ger)	C2003-256873 N(AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NI NO NZ OM PH PL PT RO RU SC SD SE SG SK SL TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW) R(AT BE BG CH CY CZ DE DK EA EE ES FI FR GB GH GM GR HU IE IT KE LS LU MC MW MZ NL OA PT RO SD SE SI SK SL SZ TR TZ UG ZM ZW)	NOVELTY 2-Propyl-heptan-1-ol alkoxylates (IA) with 3-14 ethylenoxy and 1.2-1.8 propylenoxy or pentylenoxy or 1-1.8 butylenoxy groups in the molecule are new.
Addnl. Data: RULAND A, SCHOLTISSEK M, OETTER G, TABGER K 2003.04.25 2003WO-EP04334, 2002.09.18 2002DE-1043360			<p>DETAILED DESCRIPTION</p> <p>2-Propyl-heptan-1-ol alkoxylates (IA) of formula (I), with specified numbers of alkoxylate groups, are new.</p> $  \begin{array}{c}  \text{C}_3\text{H}_7 \\    \\  \text{C}_5\text{H}_{11}-\text{C}-\text{C}-\text{O}-(\text{A})_n-(\text{CH}_2\text{CH}_2\text{O})_m-\text{H} \\    \quad   \\  \text{H} \quad \text{H}_2  \end{array}  \quad (I)  $ <p>A = propylenoxy, butylenoxy or pentylenoxy; n = 1.2-1.8 or, if A is butylenoxy, 1-1.8; and</p>
			WO 2003091191-A+

<p>m = 3-14.</p> <p><b>INDEPENDENT CLAIMS</b> are also included for the following:</p> <ol style="list-style-type: none"> <li>(1) use of a wider range of alkoxylates of formula (I); where n = 1-8; m = 2-20) as emulsifiers, foam regulators and wetting agents for hard surfaces;</li> <li>(2) preparation of alkoxylates (IA) by reacting the alkanol with propylene oxide and then with ethylene oxide under alkoxylation conditions, which can be carried out in the presence of a double metal cyanide compound as catalyst;</li> <li>(3) preparation of the alkanol by alkaline dimerization of valeraldehyde to an <math>\alpha,\beta</math>-unsaturated aldehyde, followed by hydrogenation; and</li> <li>(4) cleaning, wetting, coating, adhesive, leather degreasing, humectant, textile treatment or cosmetic, pharmaceutical or plant protection formulations containing (I).</li> </ol> <p><b>USE</b></p> <p>Alkoxylates (I) are used in surfactant formulations for cleaning hard surfaces, in humectants, cosmetic, pharmaceutical and plant protection formulations, lacquers, coating compositions, adhesives, leather degreasing agents, water treatment, the paper industry, fermentation and mineral processing and in emulsion polymerization</p>	<p>(all claimed). Their uses include humectants for the printing industry; lacquers, coating compositions, paints, pigment formulations and adhesives in the lacquer and film industries; leveling agent or yarn cleaning formulations for the textile industry; fiber processing and treatment for the paper and pulp industry; metal processing, e.g. finishing and electroplating; the food industry; water treatment and drinking water recovery; fermentation; mineral processing and dust control; building ancillaries; preparation of dispersions; and coolants and lubricants.</p> <p><b>ADVANTAGE</b></p> <p>Alkoxylates (I) have good emulsifying, foam regulation and wetting properties, with low angle of contact on hard surfaces, and reduce the surface tension of liquid systems. (I) have a favorable ecological profile, i.e. are not aquatoxic (EC 50 value over 10 mg/l for algae, daphnia or fish); are readily degradable; and are more compatible with the environment and skin than usual.</p> <p><b>EXAMPLE</b></p> <p>A mixture of 790 g 2-propylheptanol (2-PH) and 8.5 g potassium hydroxide (45% in water) in an autoclave was dried at 80°C and about</p>
	<p>WO 2003091191-A1</p>

2003-903407/82

20 mbar. To prepare alkoxylate (A), comprising 2-PH + 1.5 PO + 6 EO, 518 ml propylene oxide (PO) were added at 120-130°C and reacted completely at elevated pressure, then 1470 ml ethylene oxide (EO) were introduced at 145-155°C and reacted at elevated pressure. After purging with inert gas and cooling to room temperature, the catalyst was neutralized with 3.8 ml glacial acetic acid. Control (B), comprising 2-PH + 8 EO, was prepared by reacting 2-PH directly with 8 mole EO at 145-155°C; and control (C), comprising 2-PH + 8 EO + 1.5 PO, by reacting 2-PH first with 8 mole EO at 145-155°C and then with 1.5 mole PO at 120-130°C. The angles of contact after 0.1/10 seconds at a concentration of 0.2 g/l in water at 40°C, compared with the values for (D) demineralized water, were (A) 48/32, (B) 48/46, (C) 46/40, (D) 65/64° on V2A steel; (A) 57/40, (B) 65/64, (C) 58/54, (D) 96/95° on polyethylene; and (A) 37/20, (B) 38/25, (C) 39/24, (D) 41/39° on glass. For wetting cotton (to EN 1/52; 0.2 g/l, 2 g/l soda in distilled water) was (A) 10, (B) 15, (C) 17 seconds; and the residual PH content was (A) 0.78, (B) 4.3, (C) 1.9 g/100 g. The interfacial tension (1 g/l, 25°C, 10 minutes) was (A) 7.7, (B) 13.2, (C) 13.9 mN/m with hexadecane and (A) 5.2, (B) 8.4, (C) 8.0 with olive oil.

#### DEFINITIONS

Preferred Definitions:

n = 1.3-1.7 in (IA) and 1-6 in (I);

m = 3-12 in (IA) and 3-14 in (I); and

$C_5H_{11} = n-C_5H_{11}, C_2H_5CH(CH_3)CH_2$  and/or  $CH_3CH(CH_3)CH_2CH_2$ .  
(31pp0016DwgNo.0/0)

WO 2003091191-A/2